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Different patterns of supramolecular assembly in constitutionally similar 6-arylimidazo[2,1-*b*][1,3,4]thiadiazoles

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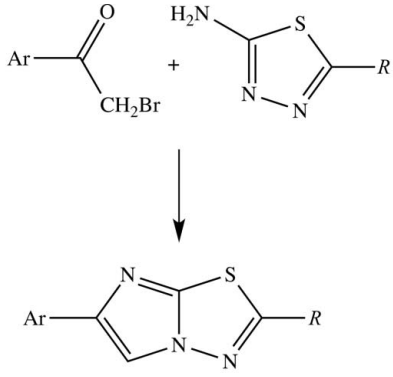
Four imidazo[2,1-*b*][1,3,4]thiadiazoles containing a simply-substituted 6-aryl group have been synthesized by reaction of 2-amino-1,3,4-thiadiazoles with bromoacetylarenes using microwave irradiation and brief reaction times. 6-(2-Chlorophenyl)imidazo[2,1-*b*][1,3,4]thiadiazole, $C_{10}H_6ClN_3S$, (I), 6-(2-chlorophenyl)-2-methylimidazo[2,1-*b*][1,3,4]thiadiazole, $C_{11}H_8ClN_3S$, (II), 6-(3,4-dichlorophenyl)imidazo[2,1-*b*][1,3,4]-thiadiazole, $C_{10}H_5Cl_2N_3S$, (III), and 6-(4-fluoro-3-methoxyphenyl)-2-methylimidazo[2,1-*b*][1,3,4]thiadiazole, $C_{12}H_{10}FN_3OS$, (IV), crystallize with Z' values of 2, 1, 1 and 2 respectively. The molecular skeletons are all nearly planar and the dihedral angles between the imidazole and aryl rings are 1.51 (8) and 7.28 (8)° in (I), 9.65 (7)° in (II), 10.44 (8)° in (III), and 1.05 (8) and 7.21 (8)° in (IV). The molecules in (I) are linked by three independent C—H···N hydrogen bonds to form ribbons containing alternating $R_2^2(8)$ and $R_4^4(18)$ rings, and these ribbons are linked into a three-dimensional array by three independent π-stacking interactions. Both (II) and (III) contain centrosymmetric dimers formed by π-stacking interactions but hydrogen bonds are absent, and the molecules of (IV) are linked into centrosymmetric $R_2^2(8)$ dimers by C—H···N hydrogen bonds. Comparisons are made with a number of related compounds.

Keywords: crystal structure; imidazo[2,1-*b*][1,3,4]thiadiazoles; molecular conformation; supramolecular assembly; pharmaceutical compounds; hydrogen bonding.

1. Introduction

Compounds containing the imidazo[2,1-*b*][1,3,4]thiadiazole unit exhibit a wide range of biological activity, including antimicrobial (Ravi *et al.*, 2009), antitubercular (Andanappa *et al.*, 2004; Shankar *et al.*, 2012) and anti-inflamatory (Jadhav *et al.*, 2008) activities. Imidazo[2,1-*b*][1,3,4]thiadiazole derivatives also act as cyclooxygenase inhibitors (Andanappa *et al.*, 2008) and antihyperlipidemic agents (Jadhav *et al.*, 2008). We report here the synthesis, molecular structures and supramolecular assembly of four related 6-arylimidazo[2,1-*b*]-[1,3,4]thiadiazoles, namely 6-(2-chlorophenyl)imidazo[2,1-*b*]-[1,3,4]thiadiazole, (I), 6-(2-chlorophenyl)-2-methylimidazo-[2,1-*b*][1,3,4]thiadiazole, (II), 6-(3,4-dichlorophenyl)imidazo-[2,1-*b*][1,3,4]thiadiazole, (III), and 6-(4-fluoro-3-methoxyphenyl)-2-methylimidazo[2,1-*b*][1,3,4]thiadiazole, (IV), which we compare with the related compounds (V)–(VIII) (see Scheme). The purposes of the present study are: (i) the comparison of the series of closely related molecular structures (I)–(IV); (ii) the exploration of the similarities and differences in their supramolecular assembly; (iii) the comparison of the structures of (I)–(IV) with the recently reported structures of some simple analogues, *viz.* compounds (V) (Praveen *et al.*, 2013), (VI) (Fun, Hemamalini *et al.*, 2011), (VII) (Fun, Yeap *et al.*, 2011) and (VIII) (Banu *et al.*, 2011) (see Scheme).



(I) R = H, Ar = 2-ClC_6H_4
(II) R = Me, Ar = 2-ClC_6H_4
(III) R = H, Ar = 3,4-$Cl_2C_6H_3$
(IV) R = Me, Ar = 3-MeO-4-FC_6H_3
(V) R = H, Ar = 3-ClC_6H_4
(VI) R = Me_2CHCH_2, Ar = Ph
(VII) R = Me_2CHCH_2, Ar = 4-MeOC_6H_4
(VIII) R = 4-F$C_6H_4CH_2$, Ar = 4-BrC_6H_4

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of each of compounds (I)–(IV), a mixture of the appropriately substituted bromoacetylarene (10 mmol) with either 2-amino-1,3,4-thiadiazole [for (I) and (III)] or 2-amino-5-methyl-1,3,4-thiadiazole [for (II) and (IV)] (10 mmol) in *N*,*N*-dimethylformamide (20 ml) was placed in a Pyrex glass tube and subjected to microwave irradiation at 373 K for 10 min, using a Biotage Initiator-microwave reactor fitted with a rotating stage. The reaction mixtures were allowed to cool to ambient temperature and were then poured onto crushed ice. The resulting solid products were collected by filtration and dried in air. Colourless crystals suitable for

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Table 1
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$C_{10}H_6ClN_3S$	$C_{11}H_8ClN_3S$	$C_{10}H_5Cl_2N_3S$	$C_{12}H_{10}FN_3OS$
M_r	235.69	249.71	270.13	263.29
Crystal system, space group	Triclinic, $P\overline{1}$	Orthorhombic, *Pbca*	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	200	200	200	200
a, *b*, *c* (Å)	7.5805 (4), 9.7942 (5), 13.6175 (6)	7.5567 (3), 11.4589 (5), 24.9006 (11)	5.5186 (2), 7.5194 (3), 12.8406 (5)	8.6766 (3), 11.7888 (4), 12.5227 (4)
α, β, γ (°)	97.712 (2), 96.549 (2), 99.416 (2)	90, 90, 90	102.027 (2), 91.293 (2), 98.430 (2)	103.039 (2), 95.189 (2), 110.365 (2)
V (Å^3)	978.77 (8)	2156.18 (16)	514.74 (3)	1149.69 (7)
Z	4	8	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.57	0.52	0.80	0.29
Crystal size (mm)	0.39 × 0.37 × 0.29	0.59 × 0.47 × 0.27	0.39 × 0.38 × 0.30	0.51 × 0.49 × 0.16
Data collection				
Diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.779, 0.851	0.773, 0.870	0.747, 0.794	0.761, 0.957
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16930, 4729, 4168	15450, 2666, 2356	12326, 2453, 2254	19758, 5695, 4829
R_{int}	0.026	0.015	0.017	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668	0.667	0.667	0.668
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.094, 1.03	0.032, 0.086, 1.06	0.027, 0.071, 1.12	0.036, 0.105, 1.05
No. of reflections	4729	2666	2453	5695
No. of parameters	271	146	145	329
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.32	0.31, −0.42	0.36, −0.33	0.38, −0.24

Computer programs: *APEX2* (Bruker, 2010), *SAINT* (Bruker, 2010), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2014) and *PLATON* (Spek, 2009).

single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in ethyl acetate. Data for compound (I): yield 69%, m.p. 448–450 K; ^{1}H NMR [dimethyl sulfoxide (DMSO)-d_6]: δ 7.33 (*m*, 1H, aryl), 7.42 (*m*, 1H, aryl), 7.53 (*d*, *J* = 7.92 Hz, 1H, aryl), 8.11 (*d*, *J* = 7.60 Hz, 1H, aryl), 8.60 (*s*, 1H, imidazole), 9.27 (*s*, 1H, thiadiazole); MS: 236 $(M + 1)^+$ for $C_{10}H_6{}^{35}ClN_3S$. Data for compound (II): yield 63%, m.p. 398–400 K; ^{1}H NMR (DMSO-d_6): δ 2.73 (*s*, 3H, methyl), 7.31 (*m*, 1H, aryl), 7.41 (*m*, 1H, aryl), 7.52 (*d*, *J* = 7.80 Hz, 1H, aryl), 8.09 (*d*, *J* = 7.84 Hz, 1H, aryl), 8.71 (*s*, 1H, imidazole); MS: 249 = $(M + 1)^+$ for $C_{11}H_8{}^{35}ClN_3S$. Data for compound (III): yield 75%, m.p. 418–419 K; ^{1}H NMR (DMSO-d_6): δ 7.66 (*d*, *J* = 8.40 Hz, 1H, aryl), 7.84 (*d*, *J* = 8.40 Hz, 1H, aryl), 8.09 (*s*, 1H, aryl), 8.86 (*s*, 1H, imidazole); MS: 271 $(M + 2)^+$ for $C_{10}H_5{}^{35}Cl_2N_3S$. Data for compound (IV): yield 76%, m.p. 375–378 K; ^{1}H NMR (DMSO-d_6): δ 2.73 (*s*, 3H, methyl), 3.90 (*s*, 3H, methoxy), 7.24 (*m*, 1H, aryl), 7.42 (*m*, 1H, aryl), 7.62 (*m*, 1H, aryl), 8.64 (*s*, 1H, imidazole); MS: 264 $(M + 1)^+$ for $C_{12}H_{10}FN_3OS$.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aryl and heterocyclic) or 0.98 Å (methyl) and with $U_{iso}(H) = kU_{eq}(C)$, where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. For compound (IV), two low-angle reflections, *viz.* $\overline{1}01$ and $\overline{1}11$, which had been attenuated by the beam stop, were omitted from the final refinements. Examination of the refined structures using *PLATON* (Spek, 2009) showed that none of them contained any solvent-accessible voids.

3. Results and discussion

The compounds reported here were all prepared using a cyclocondensation reaction between 2-amino-1,3,4-thiadiazole and a bromoacetylarene mediated by microwave irradiation (*cf.* Scheme). Exactly the same type of microwave-induced reaction had been used in the preparation of compound (V). The same type of cyclocondensation was used in the syntheses of compounds (VI)–(VIII) but, instead of using microwave irradiation, the reaction mixtures were heated under reflux in ethanol solutions, for periods of 4 h for each of (VI) and (VII) and of 18 h for (VIII). Unfortunately, no yields were reported for compounds (VI)–(VIII), but the extended reaction times used in their preparations certainly point to the efficacy of the microvave-induced syntheses for compounds (I)–(V).

The crystallization characteristics of compounds (I)–(IV) (Figs. 1–4) show some interesting and unexpected features. Although the molecular constitutions of compounds (I) and (II) differ only in the presence of a methyl group in (II) which

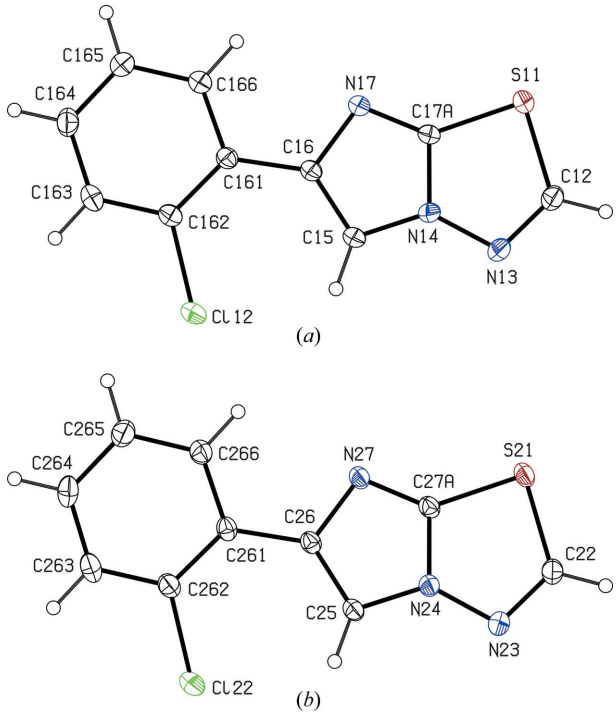

Figure 1
The structures of the two independent molecules in compound (I), showing the atom-labelling scheme for (*a*) a type 1 molecule and (*b*) a type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level.

is absent from (I), compound (I) crystallizes in the space group $P\overline{1}$ with $Z' = 2$, while compound (II) crystallizes in the space group *Pbca* with $Z' = 1$. Again, the constitutions of compounds (I) and (III) differ only in the number and locations of the chloro substituents in the aryl ring, but these compounds, although crystallizing in the same space group, do so with Z' values of 2 and 1, respectively. Compound (IV) also crystallizes with $Z' = 2$. By contrast, compound (V), which differs from (I) only in the location of the single chloro substituent, crystallizes in the monoclinic space group $P2_1/n$ with $Z' = 1$ (Praveen *et al.*, 2013). Hence, no two of the simple chloroaryl derivatives (I)–(III) and (V) are isomorphous. For compounds (I) and (IV), it will be convenient to refer to the molecules containing atoms S11 and S21 (Figs. 1 and 4) as types 1 and 2, respectively.

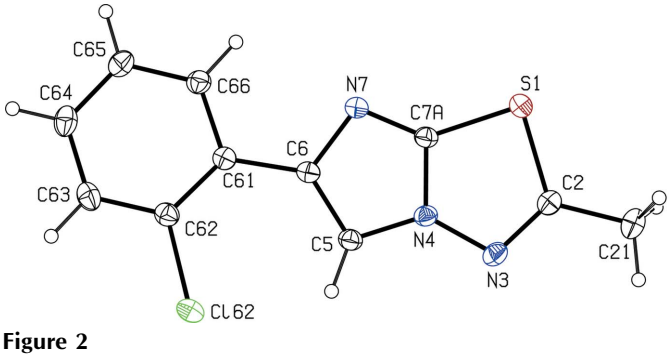

Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

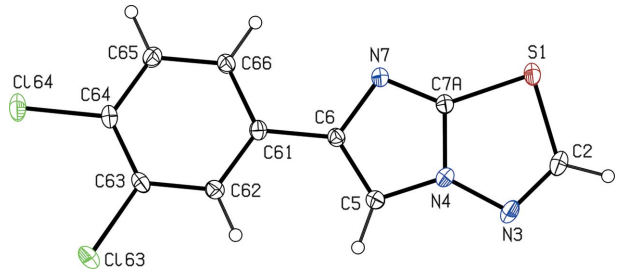

Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The bond distances in the molecules of compounds (I)–(IV) (Table 2) present some interesting patterns. The bond S*x*1—C*x*2 (where *x* = 1, 2 or nil; see Table 2 for definitions) is consistently longer than S*x*1—C*x*7*A*, regardless of whether or not there is a methyl substituent at atom C*x*2; the C*x*2—N*x*3 bond is always the shortest C—N bond in the molecule, and it may be regarded as a fully localized double bond. Of the four independent N—C bonds in the imidazole ring, N*x*7—C*x*7*A* is always significantly shorter than the other three such bonds, which have fairly similar lengths, suggesting a considerable degree of bond fixation in this ring. On the other hand, in each of compounds (I)–(III) there are close intermolecular contacts

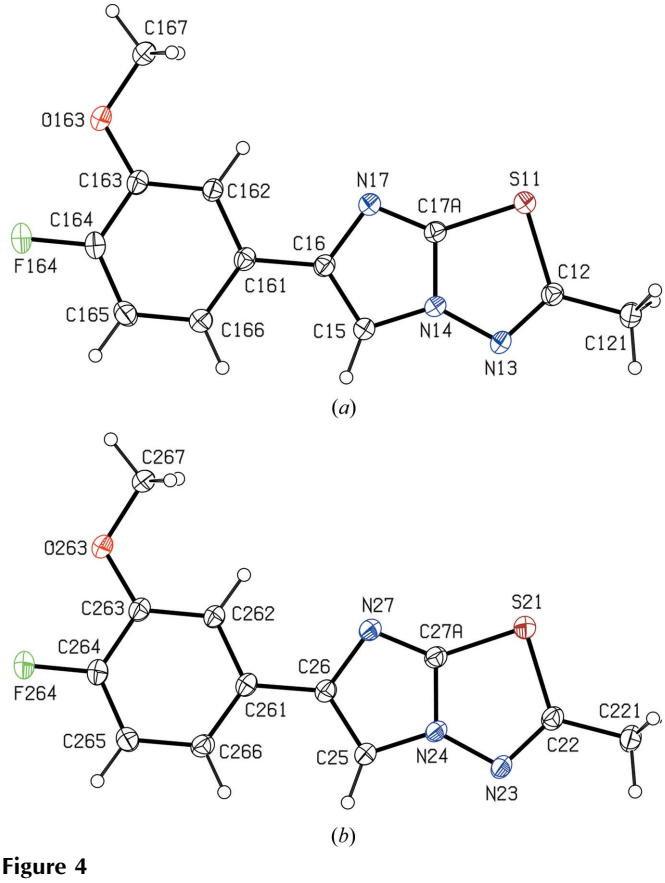

Figure 4
The structures of the two independent molecules in compound (IV), showing the atom-labelling scheme for (*a*) a type 1 molecule and (*b*) a type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Selected geometric parameters (Å, °) for compounds (I)–(IV).

Parameter	(I), molecule 1 (x = 1)	(I), molecule 2 (x = 2)	(II) (x = nil)	(III) (x = nil)	(IV), molecule 1 (x = 1)	(IV), molecule 2 (x = 2)
Sx1—Cx2	1.7441 (16)	1.7504 (16)	1.7491 (15)	1.7419 (16)	1.7605 (14)	1.7531 (15)
Cx2—Nx3	1.288 (2)	1.290 (2)	1.2937 (19)	1.288 (2)	1.2970 (18)	1.3004 (19)
Nx3—Nx4	1.3703 (17)	1.3717 (17)	1.3731 (16)	1.3730 (16)	1.3717 (15)	1.3708 (15)
Nx4—Cx5	1.3693 (19)	1.3742 (18)	1.3686 (17)	1.3706 (18)	1.3687 (17)	1.3693 (18)
Cx5—Cx6	1.379 (2)	1.378 (2)	1.3777 (19)	1.3787 (18)	1.3740 (19)	1.3759 (18)
Cx6—Nx7	1.3997 (18)	1.4018 (17)	1.3957 (17)	1.3918 (17)	1.3978 (17)	1.3940 (18)
Nx7—Cx7A	1.3093 (18)	1.3087 (19)	1.3082 (17)	1.3105 (17)	1.3075 (18)	1.3111 (17)
Cx7A—Sx1	1.7287 (15)	1.7333 (14)	1.7283 (14)	1.7310 (14)	1.7297 (14)	1.7307 (15)
Nx4—Cx7A	1.3594 (18)	1.3575 (18)	1.3583 (17)	1.3683 (18)	1.3678 (18)	1.3651 (18)
Cx63—Ox63—Cx67					116.49 (11)	116.68 (11)
Cx62—Cx63—Ox63					125.54 (13)	125.73 (13)
Cx64—Cx63—Ox63					116.19 (12)	116.04 (12)
Cx62—Cx63—Ox63—Cx67					−3.5 (2)	−10.5 (2)
Aryl/imidazole	1.51 (8)	7.28 (8)	9.65 (7)	10.44 (8)	1.05 (8)	7.21 (8)

between inversion-related pairs of imidazole rings, as discussed in detail below, and these contacts are strongly suggestive of a π–π stacking interaction in each case. A similar contact is present in the structure of (V).

The molecular skeletons in compounds (I)–(IV) are all close to planarity, as shown by the very small dihedral angles (Table 2) between the planes of the aryl and imidazole rings. The corresponding dihedral angles in compounds (V), (VII) and (VIII) are also small [6.24 (11), 4.63 (7) and 8.62 (18)°, respectively], although that in compound (VI) is rather larger [24.36 (7)°]. In compound (IV), the torsion angles Cx62—Cx63—Ox63—Cx67 (Table 2) show that the C atom of the methoxy group is close to the plane of the adjacent aryl ring; the displacements of atoms C167 and C267 from the planes of the C161–C166 and C261–C266 rings are 0.054 (2) and 0.231 (2) Å, respectively. Consistent with this, the two exocyclic C—C—O angles in each of the independent molecules of (IV) differ by almost 10°, as is typically found in planar methoxyaryl systems (Seip & Seip, 1973; Ferguson *et al.*, 1996). The C—O—C angles are both significantly larger than the near-tetrahedral value of 111.5 (15)° observed in dimethyl ether (Kimura & Kubo, 1959). Entirely comparable C—C—O and C—O—C angles are found in the structure of compound (VII), although this was not mentioned in the original structure report (Fun, Yeap *et al.*, 2011).

Although compound (I) has the simplest molecular constitution amongst the compounds reported here, it exhibits the most elaborate supramolecular assembly, involving both hydrogen bonds (Table 3) and close π–π interactions involving the imidazole rings. Two further C—H···N hydrogen bonds link the molecules into a ribbon running parallel to the [1$\bar{1}$0] direction and containing two types of centrosymmetric ring, in which $R_2^2(8)$ (Bernstein *et al.*, 1995) rings built from type 2 molecules only and centred at (n, $1-n$, $\frac{1}{2}$) alternate with $R_4^4(18)$ rings containing both types of molecule and centred at ($\frac{1}{2}+n$, $\frac{1}{2}-n$, $\frac{1}{2}$), where n represents an integer in each case (Fig. 5). Only one of these ribbons passes through each unit cell, but adjacent ribbons are linked by three independent π–π stacking interactions to form a three-dimensional array, whose formation can be analysed easily in terms of simple substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000).

Two of these interactions, involving only the type 1 molecules, link the ribbons into sheets. The imidazole rings of the

Table 3
Hydrogen bonds and short intramolecular contacts (Å, °) for compounds (I)–(IV).

*Cg*1 represents the centroid of the C61–C66 ring.

Compound	D—H···A	D—H	H···A	D···A	D—H···A
(I)	C12—H12···N27	0.95	2.33	3.280 (2)	178
	C25—H25···N23^{i}	0.95	2.61	3.480 (2)	153
	C263—H263···N13^{ii}	0.95	2.51	3.456 (2)	179
(II)	C64—H64···*Cg*1^{iii}	0.95	2.90	3.6297 (16)	135
(IV)	C15—H15···N13^{ii}	0.95	2.61	3.551 (2)	169

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $x-\frac{1}{2}, y, -z+\frac{3}{2}$.

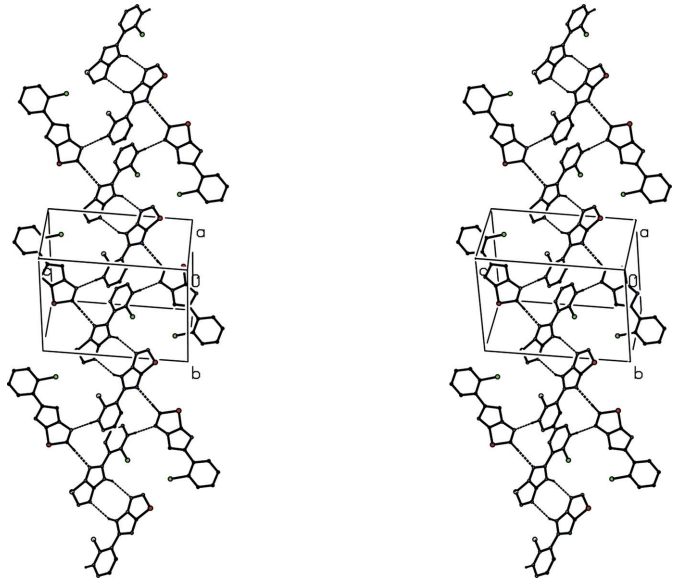

Figure 5
A stereoview of part of the crystal structure of compound (I), showing the formation of a ribbon of hydrogen-bonded $R_2^2(8)$ and $R_4^4(18)$ rings running parallel to the [1$\bar{1}$0] direction. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

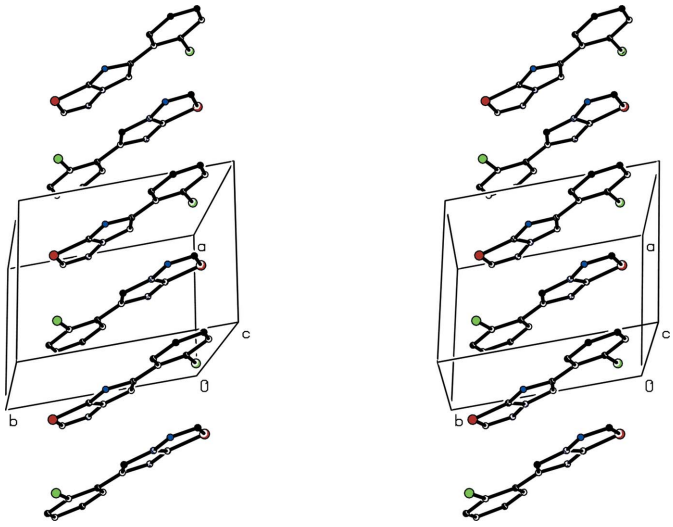

Figure 6
A stereoview of part of the crystal structure of compound (I), showing the formation of a π-stacked chain of type 1 molecules running parallel to the [100] direction and built from type 1 molecules only. For the sake of clarity, all H atoms have been omitted.

type 1 molecules at (x, y, z) and ($-x+1$, $-y+1$, $-z+2$) are parallel, with an interplanar spacing of 3.4770 (6) Å and a ring-centroid separation of 3.6542 (9) Å, corresponding to a ring-centroid offset of 1.124 Å. The imidazole ring of the type 1 molecule at (x, y, z) makes a dihedral angle of only 1.51 (8)° with the aryl ring of the type 1 molecule at ($-x+2$, $-y+1$, $-z+2$); the ring-centroid separation is 3.7460 (9) Å and the shortest perpendicular distances from the centroid of one ring to the plane of the other is 3.4542 (7) Å, corresponding to a ring-centroid offset of *ca* 1.45 Å. The combination of these

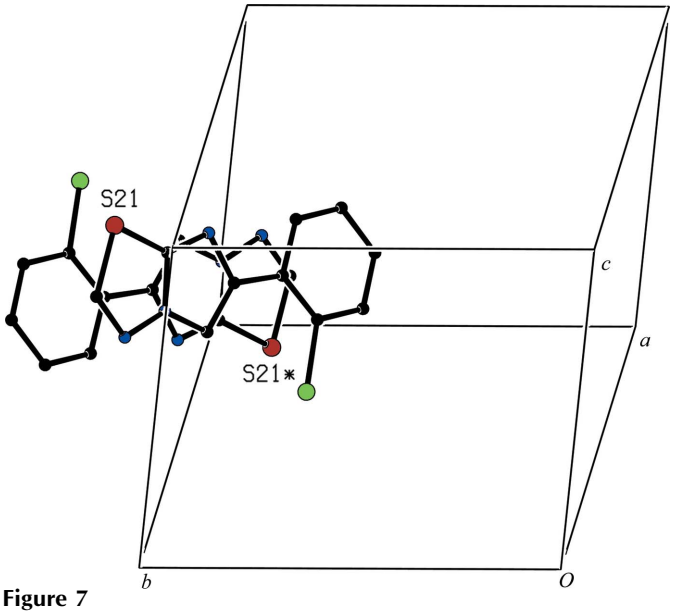

Figure 7
Part of the crystal structure of compound (I), showing the formation of a centrosymmetric π-stacked dimer centred at ($\frac{1}{2}$, 1, $\frac{1}{2}$) which links the (001) sheets. For the sake of clarity, all H atoms have been omitted and atoms marked with an asterisk (*) are at the symmetry position ($-x+1$, $-y+2$, $-z+1$).

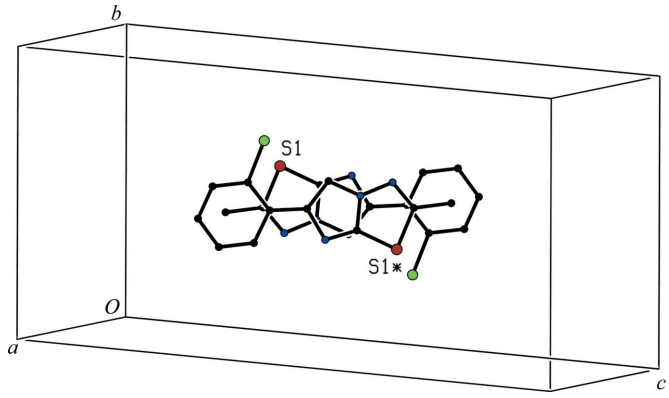

Figure 8
Part of the crystal structure of compound (II), showing the formation of a centrosymmetric π-stacked dimer centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). For the sake of clarity, all H atoms have been omitted and the S atom marked with an asterisk (*) is at the symmetry position ($-x+1$, $-y+1$, $-z+1$).

two stacking interactions generates a chain running parallel to the [100] direction (Fig. 6) which links the hydrogen-bonded ribbons to form a sheet lying parallel to (001). The third π-stacking interaction involves only type 2 molecules. The imidazole ring of the type 2 molecule at (x, y, z) makes a dihedral angle of 7.28 (8)° with the aryl ring of the type 2 molecule at ($-x+1$, $-y+2$, $-z+1$); the ring-centroid separation is 3.7159 (9) Å and the shortest perpendicular distances from the centroid of one ring to the plane of the other is 3.3905 (7) Å, corresponding to a ring-centroid offset of *ca* 1.52 Å (Fig. 7). The effect of this final π-stacking interaction is to link adjacent (001) sheets, so forming a three-dimensional structure.

The supramolecular assembly in compounds (II) and (III) is very much simpler than that in compound (I). The structure of (II) contains one short intermolecular C—H$\cdots\pi$(arene) contact (Table 3), but the H$\cdots$*Cg* and C$\cdots$*Cg* distances are both quite long and the C—H$\cdots$*Cg* angle is less than 140° so that this contact is probably not structurally significant (*cf.* Wood *et al.*, 2009), while there are no hydrogen bonds of any kind in the structure of (III). In both structures, there is a single π-stacking interaction between inversion-related pairs

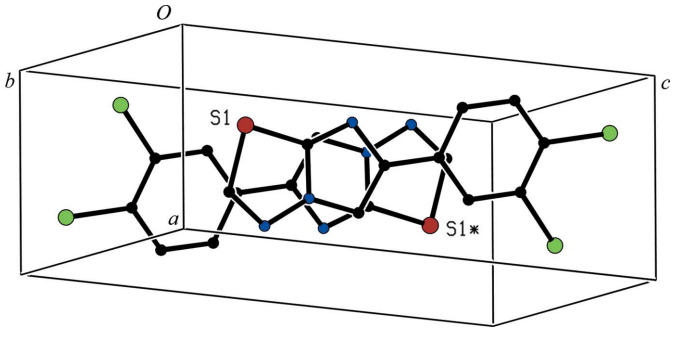

Figure 9
Part of the crystal structure of compound (III), showing the formation of a centrosymmetric π-stacked dimer centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). For the sake of clarity, all H atoms have been omitted and the S atom marked with an asterisk (*) is at the symmetry position ($-x+1$, $-y+1$, $-z+1$).

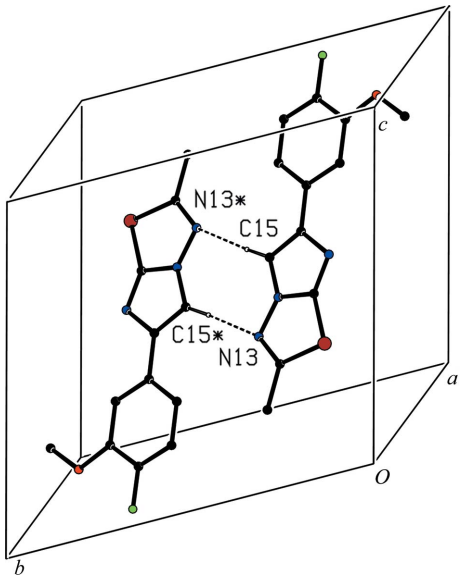

Figure 10
Part of the crystal structure of compound (IV), showing the formation of a centrosymmetric hydrogen-bonded $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. For the sake of clarity, H atoms not involved in the motif shown have been omitted and atoms marked with an asterisk (*) are at the symmetry position $(-x + 1, -y + 1, -z + 1)$.

of imidazole rings forming centrosymmetric dimers, centred in each case at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Figs. 8 and 9). In (II), the interplanar spacing is 3.4635 (6) Å, the ring-centroid separation is 3.6112 (8) Å and the ring-centroid offset is 1.022 Å; in (III), these values are 3.5296 (6) Å, 3.6696 (8)° and 1.004 Å, respectively.

In the structure of compound (IV), inversion-related pairs of type 1 molecules are linked by symmetry-related C—H···N hydrogen bonds (Table 3) to form a cyclic centrosymmetric dimer characterized by an $R_2^2(8)$ motif (Fig. 10). The only other short intermolecular contacts involve C—H bonds in methyl groups. However, when groups of approximate local C_2 and C_3 symmetry are linked by a single bond, as here, the barrier to rotation about the linking bond is very low, only a few J mol^{-1} (Tannenbaum *et al.*, 1956; Naylor & Wilson, 1957), and in such circumstances hydrocarbyl substituents generally undergo very rapid rotation around the bond linking them to the adjacent planar unit, even in the solid state (Riddell & Rogerson, 1996, 1997). Hence such contacts cannot be regarded as structurally significant. In addition to the C—H···N hydrogen bond which generates a dimer of type 1 molecules, there is a further fairly short intermolecular contact, this time between the two molecules within the selected asymmetric unit. The planes of the imidazole rings of these two molecules make a dihedral angle of only 5.712 (9)°, but the ring-centroid separation is long [3.9051 (10) Å]. The shortest perpendicular distance from the centroid of one ring to the plane of the other is quite short [3.4074 (7) Å], corresponding to a ring-centroid offset of 1.91 Å, which is probably too long for this contact to be regarded as structurally significant.

The supramolecular assembly in compounds (I)–(IV) reported here may be compared with that in compounds (V)–(VIII) (see Scheme). In compound (V) (Praveen *et al.*, 2013), there are no hydrogen bonds of any kind, but inversion-related pairs of molecules are linked by a π-stacking interaction involving the imidazole rings, exactly comparable to compounds (II) and (III), but in the structure of compound (VI) (Fun, Hemamalini *et al.*, 2011) there are neither hydrogen bonds nor π-stacking interactions. The supramolecular assembly in compound (VII) (Fun, Yeap *et al.*, 2011) is more complex than that of (V) or (VI). Inversion-related pairs of molecules are linked into centrosymmetric dimers by symmetry-related pairs of C—H···π(arene) hydrogen bonds and these dimers are linked by an aromatic π–π stacking interaction involving inversion-related pairs of aryl rings, so forming a chain of π-stacked hydrogen-bonded dimers running parallel to the [010] direction (Fig. 11), although this chain formation was not described in the original structure report. Finally, in compound (VIII (Banu *et al.*, 2011), molecules related by a 2_1 screw axis are linked by a C—H···N hydrogen bond to form a $C(6)$ chain running parallel to the [010] direction.

Thus, across the entire series of compounds (I)–(VIII), rather similar molecular constitutions are associated with a wide variety of supramolecular assembly patterns, ranging from isolated molecules in compound (VI), *via* π-stacked dimers in compounds (II), (III) and (V) and hydrogen-bonded dimers in compound (IV), to simple hydrogen-bonded chains in compound (VIII) and chains of π-stacked hydrogen-bonded dimers in compound (VII), to a three-dimensional array of π-stacked hydrogen-bonded ribbons in compound (I).

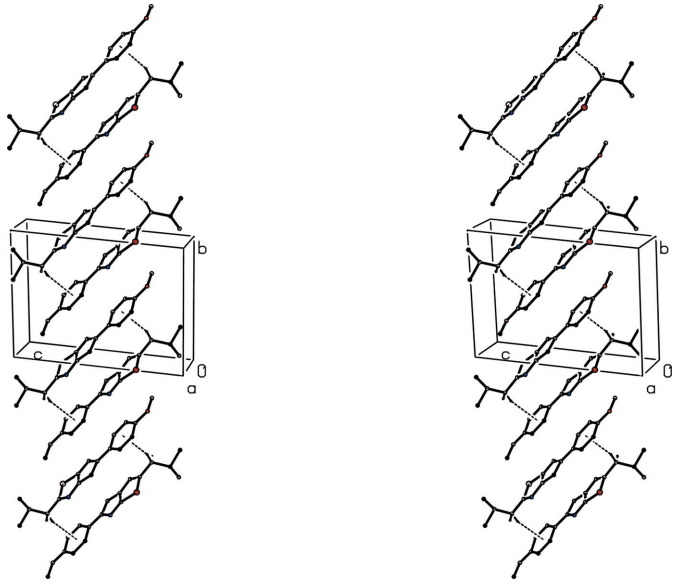

Figure 11
A stereoview of part of the crystal structure of compound (VII), showing the formation of a π-stacked chain of hydrogen-bonded dimers running parallel to the [010] direction. The original atomic coordinates (Fun, Yeap *et al.*, 2011) have been used. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, H atoms bonded to C atoms which are not involved in the motif shown have been omitted.

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Different patterns of supramolecular assembly in constitutionally similar 6-aryl-imidazo[2,1-*b*][1,3,4]thiadiazoles

Aletti S. Praveen, Hemmige S. Yathirajan, Manpreet Kaur, Badiadka Narayana, Eric C. Hosten, Richard Betz and Christopher Glidewell

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT* (Bruker, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: *PLATON* (Spek, 2009). Software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2014) and *PLATON* (Spek, 2009) for (I), (III); *SHELXL2014* (Sheldrick, 2014 and *PLATON* (Spek, 2009) for (II); *SHELXL2014* (Sheldrick, 2014) and *PLATON*(Spek, 2009 for (IV).

(I) 6-(2-Chlorophenyl)imidazo[2,1-*b*][1,3,4]thiadiazole

Crystal data

$C_{10}H_6ClN_3S$
M_r = 235.69
Triclinic, $P\overline{1}$
a = 7.5805 (4) Å
b = 9.7942 (5) Å
c = 13.6175 (6) Å
α = 97.712 (2)°
β = 96.549 (2)°
γ = 99.416 (2)°
V = 978.77 (8) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.599 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4729 reflections
θ = 1.5–28.3°
μ = 0.57 mm^{-1}
T = 200 K
Block, colourless
0.39 × 0.37 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.779, T_{max} = 0.851
16930 measured reflections
4729 independent reflections
4168 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.3°, θ_{min} = 1.5°
h = −7→10
k = −12→12
l = −14→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.094
S = 1.03
4729 reflections
271 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.4007P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S11	0.63036 (5)	0.83031 (4)	0.95902 (3)	0.03096 (10)
C12	0.6159 (2)	0.77634 (17)	0.83053 (12)	0.0321 (3)
H12	0.5851	0.8354	0.7838	0.039*
N13	0.64851 (19)	0.65344 (14)	0.80129 (9)	0.0324 (3)
N14	0.68820 (17)	0.59455 (13)	0.88501 (9)	0.0257 (3)
C15	0.7354 (2)	0.46903 (15)	0.89990 (11)	0.0274 (3)
H15	0.7490	0.3944	0.8507	0.033*
C16	0.75892 (18)	0.47526 (14)	1.00245 (10)	0.0234 (3)
N17	0.72793 (17)	0.60324 (12)	1.05002 (9)	0.0261 (3)
C17A	0.68590 (19)	0.67001 (14)	0.97609 (10)	0.0244 (3)
C161	0.81017 (18)	0.37360 (14)	1.06587 (10)	0.0240 (3)
C162	0.84856 (19)	0.24170 (15)	1.03223 (11)	0.0265 (3)
Cl12	0.82721 (6)	0.17517 (4)	0.90520 (3)	0.03711 (11)
C163	0.9007 (2)	0.15413 (16)	1.09785 (13)	0.0312 (3)
H163	0.9274	0.0658	1.0725	0.037*
C164	0.9139 (2)	0.19499 (17)	1.19985 (13)	0.0363 (4)
H164	0.9511	0.1358	1.2450	0.044*
C165	0.8722 (3)	0.32295 (19)	1.23553 (13)	0.0400 (4)
H165	0.8784	0.3512	1.3055	0.048*
C166	0.8218 (2)	0.40998 (17)	1.16984 (12)	0.0342 (3)
H166	0.7939	0.4976	1.1960	0.041*
S21	0.36631 (5)	1.18560 (4)	0.79107 (3)	0.03005 (10)
C22	0.1501 (2)	1.18596 (16)	0.73233 (11)	0.0296 (3)
H22	0.0765	1.2461	0.7606	0.036*
N23	0.09280 (17)	1.10206 (14)	0.64925 (10)	0.0305 (3)
N24	0.22771 (16)	1.02941 (13)	0.62857 (9)	0.0248 (2)
C25	0.2429 (2)	0.92574 (15)	0.55299 (10)	0.0265 (3)
H25	0.1572	0.8854	0.4958	0.032*
C26	0.41060 (19)	0.89356 (14)	0.57898 (10)	0.0237 (3)
N27	0.49777 (17)	0.97618 (13)	0.66889 (9)	0.0265 (3)
C27A	0.38205 (19)	1.05534 (15)	0.69457 (10)	0.0248 (3)
C261	0.5077 (2)	0.79204 (15)	0.52924 (10)	0.0248 (3)
C262	0.4367 (2)	0.69015 (16)	0.44566 (11)	0.0293 (3)
Cl22	0.21459 (6)	0.67224 (5)	0.38873 (4)	0.04887 (14)
C263	0.5365 (2)	0.59638 (17)	0.40445 (12)	0.0355 (3)
H263	0.4838	0.5282	0.3479	0.043*

C264	0.7127 (2)	0.60240 (18)	0.44583 (13)	0.0373 (4)
H264	0.7819	0.5384	0.4179	0.045*
C265	0.7884 (2)	0.70205 (18)	0.52824 (13)	0.0358 (3)
H265	0.9098	0.7065	0.5570	0.043*
C266	0.6875 (2)	0.79502 (16)	0.56860 (11)	0.0302 (3)
H266	0.7416	0.8632	0.6249	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.0410 (2)	0.02360 (18)	0.02883 (19)	0.01038 (15)	0.00159 (15)	0.00342 (14)
C12	0.0367 (8)	0.0318 (8)	0.0274 (7)	0.0064 (6)	−0.0010 (6)	0.0076 (6)
N13	0.0414 (7)	0.0316 (7)	0.0231 (6)	0.0065 (5)	−0.0017 (5)	0.0054 (5)
N14	0.0317 (6)	0.0238 (6)	0.0198 (5)	0.0038 (5)	0.0007 (5)	0.0009 (4)
C15	0.0350 (7)	0.0208 (6)	0.0249 (7)	0.0052 (5)	0.0030 (6)	−0.0007 (5)
C16	0.0237 (6)	0.0202 (6)	0.0250 (6)	0.0029 (5)	0.0028 (5)	0.0010 (5)
N17	0.0327 (6)	0.0221 (6)	0.0234 (6)	0.0066 (5)	0.0036 (5)	0.0015 (5)
C17A	0.0277 (7)	0.0214 (6)	0.0226 (6)	0.0042 (5)	0.0025 (5)	−0.0009 (5)
C161	0.0242 (6)	0.0213 (6)	0.0254 (7)	0.0024 (5)	0.0027 (5)	0.0023 (5)
C162	0.0257 (6)	0.0233 (7)	0.0293 (7)	0.0032 (5)	0.0052 (5)	0.0010 (5)
Cl12	0.0531 (2)	0.02745 (19)	0.0321 (2)	0.01368 (16)	0.00953 (17)	−0.00129 (15)
C163	0.0302 (7)	0.0229 (7)	0.0409 (8)	0.0067 (5)	0.0041 (6)	0.0049 (6)
C164	0.0419 (9)	0.0303 (8)	0.0387 (9)	0.0086 (7)	0.0025 (7)	0.0127 (7)
C165	0.0588 (11)	0.0348 (9)	0.0273 (8)	0.0123 (8)	0.0027 (7)	0.0056 (7)
C166	0.0485 (9)	0.0262 (7)	0.0281 (7)	0.0105 (6)	0.0041 (7)	0.0019 (6)
S21	0.0380 (2)	0.02969 (19)	0.02121 (17)	0.01047 (15)	0.00104 (14)	−0.00321 (14)
C22	0.0337 (7)	0.0287 (7)	0.0281 (7)	0.0105 (6)	0.0064 (6)	0.0026 (6)
N23	0.0296 (6)	0.0319 (7)	0.0305 (6)	0.0111 (5)	0.0040 (5)	0.0006 (5)
N24	0.0273 (6)	0.0255 (6)	0.0218 (5)	0.0077 (5)	0.0022 (5)	0.0015 (5)
C25	0.0302 (7)	0.0264 (7)	0.0213 (6)	0.0064 (5)	0.0003 (5)	−0.0013 (5)
C26	0.0300 (7)	0.0221 (6)	0.0187 (6)	0.0054 (5)	0.0021 (5)	0.0029 (5)
N27	0.0318 (6)	0.0265 (6)	0.0206 (5)	0.0092 (5)	0.0002 (5)	−0.0003 (5)
C27A	0.0303 (7)	0.0248 (7)	0.0189 (6)	0.0066 (5)	0.0007 (5)	0.0019 (5)
C261	0.0324 (7)	0.0226 (6)	0.0206 (6)	0.0072 (5)	0.0051 (5)	0.0036 (5)
C262	0.0367 (8)	0.0263 (7)	0.0240 (7)	0.0074 (6)	0.0007 (6)	0.0010 (5)
Cl22	0.0461 (2)	0.0448 (2)	0.0458 (2)	0.01490 (19)	−0.01480 (19)	−0.01819 (19)
C263	0.0499 (9)	0.0288 (8)	0.0278 (7)	0.0107 (7)	0.0075 (7)	−0.0019 (6)
C264	0.0483 (9)	0.0355 (9)	0.0340 (8)	0.0186 (7)	0.0159 (7)	0.0042 (7)
C265	0.0353 (8)	0.0403 (9)	0.0353 (8)	0.0150 (7)	0.0079 (7)	0.0055 (7)
C266	0.0335 (7)	0.0318 (8)	0.0252 (7)	0.0086 (6)	0.0037 (6)	0.0013 (6)

Geometric parameters (Å, º)

S11—C17A	1.7287 (15)	S21—C27A	1.7333 (14)
S11—C12	1.7441 (16)	S21—C22	1.7405 (16)
C12—N13	1.288 (2)	C22—N23	1.290 (2)
C12—H12	0.9500	C22—H22	0.9500
N13—N14	1.3703 (17)	N23—N24	1.3717 (17)

N14—C17A	1.3594 (18)	N24—C27A	1.3575 (18)
N14—C15	1.3693 (19)	N24—C25	1.3742 (18)
C15—C16	1.379 (2)	C25—C26	1.378 (2)
C15—H15	0.9500	C25—H25	0.9500
C16—N17	1.3997 (18)	C26—N27	1.4018 (17)
C16—C161	1.4727 (19)	C26—C261	1.470 (2)
N17—C17A	1.3093 (19)	N27—C27A	1.3087 (19)
C161—C162	1.399 (2)	C261—C266	1.400 (2)
C161—C166	1.402 (2)	C261—C262	1.4003 (19)
C162—C163	1.388 (2)	C262—C263	1.384 (2)
C162—Cl12	1.7441 (15)	C262—Cl22	1.7427 (16)
C163—C164	1.380 (2)	C263—C264	1.378 (3)
C163—H163	0.9500	C263—H263	0.9500
C164—C165	1.381 (2)	C264—C265	1.385 (2)
C164—H164	0.9500	C264—H264	0.9500
C165—C166	1.381 (2)	C265—C266	1.381 (2)
C165—H165	0.9500	C265—H265	0.9500
C166—H166	0.9500	C266—H266	0.9500

C17A—S11—C12	87.72 (7)	C27A—S21—C22	87.38 (7)
N13—C12—S11	117.55 (12)	N23—C22—S21	118.02 (12)
N13—C12—H12	121.2	N23—C22—H22	121.0
S11—C12—H12	121.2	S21—C22—H22	121.0
C12—N13—N14	107.55 (12)	C22—N23—N24	107.25 (12)
C17A—N14—C15	108.02 (12)	C27A—N24—N23	118.36 (12)
C17A—N14—N13	118.43 (12)	C27A—N24—C25	107.97 (12)
C15—N14—N13	133.54 (12)	N23—N24—C25	133.63 (12)
N14—C15—C16	104.46 (12)	N24—C25—C26	104.32 (12)
N14—C15—H15	127.8	N24—C25—H25	127.8
C16—C15—H15	127.8	C26—C25—H25	127.8
C15—C16—N17	110.91 (12)	C25—C26—N27	111.03 (12)
C15—C16—C161	131.26 (13)	C25—C26—C261	131.46 (13)
N17—C16—C161	117.82 (12)	N27—C26—C261	117.51 (12)
C17A—N17—C16	103.95 (12)	C27A—N27—C26	103.84 (12)
N17—C17A—N14	112.65 (13)	N27—C27A—N24	112.83 (12)
N17—C17A—S11	138.59 (11)	N27—C27A—S21	138.21 (11)
N14—C17A—S11	108.76 (10)	N24—C27A—S21	108.96 (10)
C162—C161—C166	115.90 (13)	C266—C261—C262	116.06 (13)
C162—C161—C16	126.07 (13)	C266—C261—C26	117.91 (13)
C166—C161—C16	118.03 (13)	C262—C261—C26	126.03 (13)
C163—C162—C161	122.07 (14)	C263—C262—C261	122.42 (14)
C163—C162—Cl12	115.91 (11)	C263—C262—Cl22	116.03 (12)
C161—C162—Cl12	122.00 (11)	C261—C262—Cl22	121.54 (12)
C164—C163—C162	120.23 (15)	C264—C263—C262	119.66 (15)
C164—C163—H163	119.9	C264—C263—H263	120.2
C162—C163—H163	119.9	C262—C263—H263	120.2
C163—C164—C165	119.19 (15)	C263—C264—C265	119.79 (15)
C163—C164—H164	120.4	C263—C264—H264	120.1

C165—C164—H164	120.4	C265—C264—H264	120.1
C166—C165—C164	120.26 (16)	C266—C265—C264	119.97 (16)
C166—C165—H165	119.9	C266—C265—H265	120.0
C164—C165—H165	119.9	C264—C265—H265	120.0
C165—C166—C161	122.32 (15)	C265—C266—C261	122.09 (14)
C165—C166—H166	118.8	C265—C266—H266	119.0
C161—C166—H166	118.8	C261—C266—H266	119.0

C17A—S11—C12—N13	−0.16 (14)	C27A—S21—C22—N23	−1.15 (13)
S11—C12—N13—N14	0.07 (18)	S21—C22—N23—N24	0.34 (17)
C12—N13—N14—C17A	0.10 (19)	C22—N23—N24—C27A	1.02 (18)
C12—N13—N14—C15	−178.82 (16)	C22—N23—N24—C25	178.35 (15)
C17A—N14—C15—C16	0.34 (16)	C27A—N24—C25—C26	0.35 (16)
N13—N14—C15—C16	179.34 (15)	N23—N24—C25—C26	−177.18 (14)
N14—C15—C16—N17	−0.45 (16)	N24—C25—C26—N27	−0.17 (16)
N14—C15—C16—C161	−179.95 (14)	N24—C25—C26—C261	−179.68 (14)
C15—C16—N17—C17A	0.39 (16)	C25—C26—N27—C27A	−0.09 (16)
C161—C16—N17—C17A	179.96 (12)	C261—C26—N27—C27A	179.50 (12)
C16—N17—C17A—N14	−0.17 (16)	C26—N27—C27A—N24	0.32 (17)
C16—N17—C17A—S11	−178.84 (14)	C26—N27—C27A—S21	179.45 (14)
C15—N14—C17A—N17	−0.11 (17)	N23—N24—C27A—N27	177.53 (12)
N13—N14—C17A—N17	−179.28 (13)	C25—N24—C27A—N27	−0.44 (17)
C15—N14—C17A—S11	178.96 (10)	N23—N24—C27A—S21	−1.86 (16)
N13—N14—C17A—S11	−0.22 (16)	C25—N24—C27A—S21	−179.83 (10)
C12—S11—C17A—N17	178.89 (18)	C22—S21—C27A—N27	−177.60 (18)
C12—S11—C17A—N14	0.19 (11)	C22—S21—C27A—N24	1.55 (11)
C15—C16—C161—C162	0.3 (2)	C25—C26—C261—C266	172.61 (15)
N17—C16—C161—C162	−179.15 (13)	N27—C26—C261—C266	−6.9 (2)
C15—C16—C161—C166	−179.44 (16)	C25—C26—C261—C262	−8.0 (3)
N17—C16—C161—C166	1.10 (19)	N27—C26—C261—C262	172.46 (14)
C166—C161—C162—C163	−2.0 (2)	C266—C261—C262—C263	0.7 (2)
C16—C161—C162—C163	178.20 (14)	C26—C261—C262—C263	−178.66 (15)
C166—C161—C162—Cl12	175.99 (12)	C266—C261—C262—Cl22	179.48 (11)
C16—C161—C162—Cl12	−3.8 (2)	C26—C261—C262—Cl22	0.1 (2)
C161—C162—C163—C164	0.9 (2)	C261—C262—C263—C264	−0.4 (3)
Cl12—C162—C163—C164	−177.25 (12)	Cl22—C262—C263—C264	−179.21 (13)
C162—C163—C164—C165	0.9 (3)	C262—C263—C264—C265	0.0 (3)
C163—C164—C165—C166	−1.3 (3)	C263—C264—C265—C266	0.0 (3)
C164—C165—C166—C161	0.1 (3)	C264—C265—C266—C261	0.4 (3)
C162—C161—C166—C165	1.5 (2)	C262—C261—C266—C265	−0.7 (2)
C16—C161—C166—C165	−178.67 (16)	C26—C261—C266—C265	178.69 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···N27	0.95	2.33	3.280 (2)	178

C25—H25···N23^{i}	0.95	2.61	3.480 (2)	153
C263—H263···N13^{ii}	0.95	2.51	3.456 (2)	179

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1.

(II) 6-(2-Chlorophenyl)-2-methylimidazo[2,1-*b*][1,3,4]thiadiazole

Crystal data

$C_{11}H_8ClN_3S$
M_r = 249.71
Orthorhombic, *Pbca*
a = 7.5567 (3) Å
b = 11.4589 (5) Å
c = 24.9006 (11) Å
V = 2156.18 (16) Å^3
Z = 8
F(000) = 1024
D_x = 1.538 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2666 reflections
θ = 3.2–28.3°
μ = 0.52 mm^{-1}
T = 200 K
Block, colourless
0.59 × 0.47 × 0.27 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.773, T_{max} = 0.870
15450 measured reflections
2666 independent reflections
2356 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 28.3°, θ_{min} = 3.2°
h = −10→10
k = −11→15
l = −31→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.06
2666 reflections
146 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 1.0733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.80106 (5)	0.65665 (3)	0.45158 (2)	0.03626 (12)
C2	0.85112 (17)	0.51521 (13)	0.42998 (6)	0.0287 (3)
N3	0.81433 (16)	0.43078 (11)	0.46262 (5)	0.0312 (3)
N4	0.74098 (15)	0.47874 (10)	0.50798 (4)	0.0262 (2)
C5	0.67939 (19)	0.43257 (12)	0.55510 (5)	0.0287 (3)
H5	0.6739	0.3527	0.5652	0.034*
C6	0.62709 (16)	0.52820 (11)	0.58465 (5)	0.0243 (3)

N7	0.65651 (16)	0.63161 (10)	0.55642 (5)	0.0289 (2)
C7A	0.72392 (17)	0.59658 (12)	0.51079 (5)	0.0266 (3)
C21	0.93002 (19)	0.49516 (15)	0.37581 (6)	0.0369 (3)
H21A	0.8435	0.5158	0.3481	0.055*
H21B	0.9626	0.4128	0.3721	0.055*
H21C	1.0358	0.5438	0.3717	0.055*
C61	0.54653 (16)	0.53729 (11)	0.63833 (5)	0.0252 (3)
C62	0.52735 (19)	0.44666 (12)	0.67546 (5)	0.0297 (3)
Cl62	0.61184 (7)	0.30845 (4)	0.66226 (2)	0.05021 (14)
C63	0.4444 (2)	0.46260 (14)	0.72479 (6)	0.0352 (3)
H63	0.4322	0.3987	0.7488	0.042*
C64	0.3800 (2)	0.57038 (15)	0.73882 (6)	0.0370 (3)
H64	0.3229	0.5815	0.7724	0.044*
C65	0.3993 (2)	0.66291 (15)	0.70344 (6)	0.0359 (3)
H65	0.3564	0.7380	0.7130	0.043*
C66	0.48064 (19)	0.64626 (13)	0.65427 (5)	0.0304 (3)
H66	0.4922	0.7107	0.6305	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0470 (2)	0.02767 (19)	0.03408 (19)	−0.00163 (15)	0.01139 (15)	0.00237 (14)
C2	0.0254 (6)	0.0316 (7)	0.0291 (6)	−0.0011 (5)	−0.0019 (5)	−0.0043 (5)
N3	0.0355 (6)	0.0298 (6)	0.0285 (6)	0.0010 (5)	−0.0011 (5)	−0.0074 (5)
N4	0.0299 (5)	0.0221 (5)	0.0267 (5)	−0.0007 (4)	−0.0020 (4)	−0.0030 (4)
C5	0.0366 (7)	0.0222 (6)	0.0274 (6)	−0.0018 (5)	−0.0027 (5)	0.0005 (5)
C6	0.0243 (5)	0.0224 (6)	0.0264 (6)	−0.0006 (5)	−0.0047 (5)	0.0007 (5)
N7	0.0340 (6)	0.0227 (5)	0.0300 (6)	0.0011 (5)	0.0031 (5)	0.0010 (4)
C7A	0.0281 (6)	0.0216 (6)	0.0300 (6)	−0.0012 (5)	0.0002 (5)	0.0003 (5)
C21	0.0320 (7)	0.0484 (9)	0.0303 (7)	−0.0008 (7)	0.0018 (5)	−0.0077 (6)
C61	0.0241 (6)	0.0267 (6)	0.0247 (6)	−0.0022 (5)	−0.0056 (5)	−0.0009 (5)
C62	0.0319 (6)	0.0281 (7)	0.0292 (6)	−0.0018 (5)	−0.0037 (5)	0.0015 (5)
Cl62	0.0779 (3)	0.0297 (2)	0.0430 (2)	0.01115 (19)	0.0113 (2)	0.01119 (16)
C63	0.0384 (7)	0.0400 (8)	0.0272 (7)	−0.0071 (6)	−0.0022 (6)	0.0051 (6)
C64	0.0359 (7)	0.0488 (9)	0.0262 (7)	−0.0060 (7)	0.0005 (5)	−0.0053 (6)
C65	0.0392 (7)	0.0371 (8)	0.0315 (7)	0.0017 (6)	−0.0004 (6)	−0.0073 (6)
C66	0.0348 (7)	0.0285 (7)	0.0278 (6)	0.0007 (6)	−0.0032 (5)	−0.0014 (5)

Geometric parameters (Å, º)

S1—C7A	1.7283 (14)	C21—H21B	0.9800
S1—C2	1.7491 (15)	C21—H21C	0.9800
C2—N3	1.2937 (19)	C61—C62	1.3980 (19)
C2—C21	1.4927 (19)	C61—C66	1.4016 (19)
N3—N4	1.3731 (16)	C62—C63	1.391 (2)
N4—C7A	1.3583 (17)	C62—Cl62	1.7389 (15)
N4—C5	1.3686 (17)	C63—C64	1.372 (2)
C5—C6	1.3777 (19)	C63—H63	0.9500

C5—H5	0.9500	C64—C65	1.386 (2)
C6—N7	1.3957 (17)	C64—H64	0.9500
C6—C61	1.4725 (18)	C65—C66	1.383 (2)
N7—C7A	1.3082 (17)	C65—H65	0.9500
C21—H21A	0.9800	C66—H66	0.9500

C7A—S1—C2	88.06 (7)	C2—C21—H21C	109.5
N3—C2—C21	122.58 (13)	H21A—C21—H21C	109.5
N3—C2—S1	116.96 (11)	H21B—C21—H21C	109.5
C21—C2—S1	120.45 (11)	C62—C61—C66	115.96 (12)
C2—N3—N4	107.71 (12)	C62—C61—C6	126.20 (12)
C7A—N4—C5	107.93 (11)	C66—C61—C6	117.84 (12)
C7A—N4—N3	118.59 (11)	C63—C62—C61	122.21 (13)
C5—N4—N3	133.46 (12)	C63—C62—Cl62	116.87 (11)
N4—C5—C6	104.36 (12)	C61—C62—Cl62	120.90 (11)
N4—C5—H5	127.8	C64—C63—C62	120.16 (14)
C6—C5—H5	127.8	C64—C63—H63	119.9
C5—C6—N7	111.13 (12)	C62—C63—H63	119.9
C5—C6—C61	131.28 (12)	C63—C64—C65	119.30 (14)
N7—C6—C61	117.58 (11)	C63—C64—H64	120.3
C7A—N7—C6	103.83 (11)	C65—C64—H64	120.3
N7—C7A—N4	112.74 (12)	C66—C65—C64	120.24 (15)
N7—C7A—S1	138.59 (11)	C66—C65—H65	119.9
N4—C7A—S1	108.67 (10)	C64—C65—H65	119.9
C2—C21—H21A	109.5	C65—C66—C61	122.10 (14)
C2—C21—H21B	109.5	C65—C66—H66	119.0
H21A—C21—H21B	109.5	C61—C66—H66	119.0

C7A—S1—C2—N3	0.40 (11)	C2—S1—C7A—N7	178.65 (16)
C7A—S1—C2—C21	179.46 (12)	C2—S1—C7A—N4	−0.86 (10)
C21—C2—N3—N4	−178.84 (12)	C5—C6—C61—C62	9.8 (2)
S1—C2—N3—N4	0.20 (15)	N7—C6—C61—C62	−171.26 (13)
C2—N3—N4—C7A	−0.96 (16)	C5—C6—C61—C66	−169.56 (14)
C2—N3—N4—C5	−179.32 (14)	N7—C6—C61—C66	9.33 (17)
C7A—N4—C5—C6	−0.05 (14)	C66—C61—C62—C63	1.4 (2)
N3—N4—C5—C6	178.44 (13)	C6—C61—C62—C63	−178.00 (13)
N4—C5—C6—N7	−0.25 (15)	C66—C61—C62—Cl62	−177.10 (10)
N4—C5—C6—C61	178.70 (13)	C6—C61—C62—Cl62	3.49 (19)
C5—C6—N7—C7A	0.45 (15)	C61—C62—C63—C64	−0.9 (2)
C61—C6—N7—C7A	−178.66 (11)	Cl62—C62—C63—C64	177.67 (12)
C6—N7—C7A—N4	−0.49 (15)	C62—C63—C64—C65	−0.2 (2)
C6—N7—C7A—S1	−179.99 (13)	C63—C64—C65—C66	0.7 (2)
C5—N4—C7A—N7	0.36 (16)	C64—C65—C66—C61	−0.2 (2)
N3—N4—C7A—N7	−178.40 (11)	C62—C61—C66—C65	−0.9 (2)
C5—N4—C7A—S1	−179.99 (9)	C6—C61—C66—C65	178.58 (13)
N3—N4—C7A—S1	1.25 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C64—H64···*Cg*1	0.95	2.69	3.6297 (16)	135

(III) 6-(3,4-Dichlorophenyl)imidazo[2,1-*b*][1,3,4]thiadiazole

Crystal data

$C_{10}H_5Cl_2N_3S$
M_r = 270.13
Triclinic, $P\overline{1}$
a = 5.5186 (2) Å
b = 7.5194 (3) Å
c = 12.8406 (5) Å
α = 102.027 (2)°
β = 91.293 (2)°
γ = 98.430 (2)°
V = 514.74 (3) Å^3
Z = 2
$F(000)$ = 272
D_x = 1.743 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2453 reflections
θ = 1.6–28.3°
μ = 0.80 mm^{-1}
T = 200 K
Block, colourless
0.39 × 0.38 × 0.30 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.747, T_{max} = 0.794
12326 measured reflections
2453 independent reflections
2254 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −7→7
k = −10→9
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.071
S = 1.12
2453 reflections
145 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.2696P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.38539 (7)	0.23816 (5)	0.21457 (3)	0.02811 (10)
C2	0.6946 (3)	0.3233 (2)	0.20847 (12)	0.0309 (3)
H2	0.7601	0.3434	0.1434	0.037*
N3	0.8315 (2)	0.35812 (19)	0.29503 (11)	0.0315 (3)
N4	0.6869 (2)	0.31365 (16)	0.37429 (10)	0.0239 (2)

C5	0.7299 (3)	0.31705 (19)	0.48029 (11)	0.0246 (3)
H5	0.8808	0.3550	0.5215	0.029*
C6	0.5040 (2)	0.25266 (17)	0.51328 (10)	0.0197 (2)
N7	0.3244 (2)	0.20853 (16)	0.43029 (9)	0.0227 (2)
C7A	0.4436 (2)	0.24777 (18)	0.34882 (11)	0.0219 (3)
C61	0.4443 (2)	0.22702 (17)	0.62041 (10)	0.0203 (3)
C62	0.6141 (3)	0.29729 (18)	0.70661 (11)	0.0230 (3)
H62	0.7688	0.3634	0.6962	0.028*
C63	0.5574 (3)	0.27087 (19)	0.80769 (11)	0.0239 (3)
Cl63	0.77020 (8)	0.36636 (6)	0.91302 (3)	0.04046 (12)
C64	0.3335 (3)	0.17252 (19)	0.82416 (11)	0.0237 (3)
Cl64	0.26526 (8)	0.12731 (6)	0.94788 (3)	0.03559 (11)
C65	0.1627 (3)	0.10504 (19)	0.73917 (11)	0.0261 (3)
H65	0.0078	0.0397	0.7501	0.031*
C66	0.2170 (3)	0.13257 (18)	0.63811 (11)	0.0232 (3)
H66	0.0982	0.0867	0.5804	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0326 (2)	0.03413 (19)	0.01719 (17)	0.00065 (14)	0.00128 (14)	0.00772 (13)
C2	0.0342 (8)	0.0359 (8)	0.0253 (7)	0.0052 (6)	0.0105 (6)	0.0123 (6)
N3	0.0285 (6)	0.0405 (7)	0.0284 (7)	0.0018 (5)	0.0098 (5)	0.0158 (5)
N4	0.0220 (6)	0.0271 (6)	0.0228 (6)	0.0002 (4)	0.0043 (5)	0.0084 (4)
C5	0.0225 (6)	0.0282 (7)	0.0223 (7)	−0.0010 (5)	−0.0014 (5)	0.0076 (5)
C6	0.0216 (6)	0.0191 (6)	0.0176 (6)	0.0016 (5)	−0.0001 (5)	0.0032 (4)
N7	0.0228 (6)	0.0271 (6)	0.0177 (5)	0.0009 (4)	0.0010 (4)	0.0056 (4)
C7A	0.0231 (6)	0.0236 (6)	0.0187 (6)	0.0021 (5)	0.0010 (5)	0.0047 (5)
C61	0.0239 (6)	0.0193 (6)	0.0181 (6)	0.0044 (5)	0.0020 (5)	0.0037 (4)
C62	0.0220 (6)	0.0247 (6)	0.0211 (6)	0.0024 (5)	−0.0001 (5)	0.0031 (5)
C63	0.0263 (7)	0.0273 (7)	0.0174 (6)	0.0065 (5)	−0.0037 (5)	0.0023 (5)
Cl63	0.0346 (2)	0.0608 (3)	0.02111 (19)	−0.00011 (18)	−0.00828 (15)	0.00398 (16)
C64	0.0310 (7)	0.0255 (6)	0.0163 (6)	0.0075 (5)	0.0030 (5)	0.0062 (5)
Cl64	0.0453 (2)	0.0452 (2)	0.02001 (18)	0.00896 (17)	0.00597 (15)	0.01367 (15)
C65	0.0267 (7)	0.0268 (7)	0.0236 (7)	−0.0015 (5)	0.0031 (6)	0.0067 (5)
C66	0.0252 (7)	0.0241 (6)	0.0183 (6)	−0.0003 (5)	−0.0010 (5)	0.0035 (5)

Geometric parameters (Å, º)

S1—C7A	1.7310 (14)	C61—C62	1.3951 (19)
S1—C2	1.7419 (16)	C61—C66	1.3963 (19)
C2—N3	1.288 (2)	C62—C63	1.3891 (19)
C2—H2	0.9500	C62—H62	0.9500
N3—N4	1.3730 (16)	C63—C64	1.389 (2)
N4—C7A	1.3683 (18)	C63—Cl63	1.7319 (14)
N4—C5	1.3706 (18)	C64—C65	1.385 (2)
C5—C6	1.3787 (18)	C64—Cl64	1.7308 (13)
C5—H5	0.9500	C65—C66	1.3885 (18)

C6—N7	1.3918 (17)	C65—H65	0.9500
C6—C61	1.4664 (18)	C66—H66	0.9500
N7—C7A	1.3105 (17)		

C7A—S1—C2	87.78 (7)	C62—C61—C66	118.83 (12)
N3—C2—S1	117.87 (11)	C62—C61—C6	120.44 (12)
N3—C2—H2	121.1	C66—C61—C6	120.73 (12)
S1—C2—H2	121.1	C63—C62—C61	120.16 (13)
C2—N3—N4	107.53 (12)	C63—C62—H62	119.9
C7A—N4—C5	107.67 (11)	C61—C62—H62	119.9
C7A—N4—N3	118.21 (12)	C64—C63—C62	120.69 (13)
C5—N4—N3	134.12 (12)	C64—C63—Cl63	120.76 (11)
N4—C5—C6	104.11 (12)	C62—C63—Cl63	118.55 (11)
N4—C5—H5	127.9	C65—C64—C63	119.37 (12)
C6—C5—H5	127.9	C65—C64—Cl64	119.10 (11)
C5—C6—N7	111.90 (12)	C63—C64—Cl64	121.51 (11)
C5—C6—C61	127.32 (13)	C64—C65—C66	120.26 (13)
N7—C6—C61	120.78 (12)	C64—C65—H65	119.9
C7A—N7—C6	103.47 (11)	C66—C65—H65	119.9
N7—C7A—N4	112.85 (12)	C65—C66—C61	120.66 (13)
N7—C7A—S1	138.53 (11)	C65—C66—H66	119.7
N4—C7A—S1	108.61 (10)	C61—C66—H66	119.7

C7A—S1—C2—N3	−0.49 (13)	C5—C6—C61—C62	10.8 (2)
S1—C2—N3—N4	0.68 (17)	N7—C6—C61—C62	−170.07 (12)
C2—N3—N4—C7A	−0.59 (18)	C5—C6—C61—C66	−169.40 (13)
C2—N3—N4—C5	178.33 (15)	N7—C6—C61—C66	9.73 (19)
C7A—N4—C5—C6	−0.43 (15)	C66—C61—C62—C63	0.9 (2)
N3—N4—C5—C6	−179.44 (14)	C6—C61—C62—C63	−179.33 (12)
N4—C5—C6—N7	0.50 (15)	C61—C62—C63—C64	0.9 (2)
N4—C5—C6—C61	179.70 (12)	C61—C62—C63—Cl63	−178.12 (10)
C5—C6—N7—C7A	−0.36 (15)	C62—C63—C64—C65	−1.9 (2)
C61—C6—N7—C7A	−179.62 (12)	Cl63—C63—C64—C65	177.06 (11)
C6—N7—C7A—N4	0.07 (15)	C62—C63—C64—Cl64	176.50 (11)
C6—N7—C7A—S1	178.89 (13)	Cl63—C63—C64—Cl64	−4.52 (17)
C5—N4—C7A—N7	0.24 (16)	C63—C64—C65—C66	1.2 (2)
N3—N4—C7A—N7	179.43 (12)	Cl64—C64—C65—C66	−177.23 (11)
C5—N4—C7A—S1	−178.94 (9)	C64—C65—C66—C61	0.5 (2)
N3—N4—C7A—S1	0.25 (15)	C62—C61—C66—C65	−1.6 (2)
C2—S1—C7A—N7	−178.75 (17)	C6—C61—C66—C65	178.64 (12)
C2—S1—C7A—N4	0.11 (10)		

(IV) 6-(4-Fluoro-3-methoxyphenyl)-2-methylimidazo[2,1-*b*][1,3,4]thiadiazole

Crystal data

$C_{12}H_{10}FN_3OS$	a = 8.6766 (3) Å
M_r = 263.29	b = 11.7888 (4) Å
Triclinic, $P\overline{1}$	c = 12.5227 (4) Å

α = 103.039 (2)°
β = 95.189 (2)°
γ = 110.365 (2)°
V = 1149.69 (7) Å^3
Z = 4
F(000) = 544
D_x = 1.521 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5697 reflections
θ = 2.2–28.4°
μ = 0.29 mm^{-1}
T = 200 K
Block, colourless
0.51 × 0.49 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.761, T_{max} = 0.957
19758 measured reflections

5695 independent reflections
4829 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.4°, θ_{min} = 2.2°
h = −11→11
k = −15→15
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.105
S = 1.05
5695 reflections
329 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.3149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S11	0.00646 (4)	0.13559 (3)	0.37155 (3)	0.03362 (10)
C12	0.08673 (17)	0.27326 (13)	0.32795 (11)	0.0301 (3)
N13	0.22531 (15)	0.35907 (11)	0.38809 (9)	0.0308 (2)
N14	0.27389 (14)	0.31605 (11)	0.47316 (9)	0.0285 (2)
C15	0.40549 (18)	0.36681 (13)	0.56107 (11)	0.0308 (3)
H15	0.4947	0.4467	0.5792	0.037*
C16	0.37966 (17)	0.27620 (13)	0.61728 (11)	0.0279 (3)
N17	0.23394 (15)	0.17076 (11)	0.56618 (10)	0.0314 (3)
C17A	0.17615 (17)	0.19957 (13)	0.48028 (11)	0.0285 (3)
C121	0.00056 (19)	0.28957 (15)	0.22791 (12)	0.0373 (3)
H12A	0.0027	0.2284	0.1611	0.056*
H12B	0.0578	0.3750	0.2219	0.056*
H12C	−0.1157	0.2761	0.2348	0.056*
C161	0.48497 (17)	0.28344 (13)	0.71864 (11)	0.0289 (3)
C162	0.43884 (17)	0.18333 (13)	0.76701 (11)	0.0289 (3)

H162	0.3399	0.1112	0.7334	0.035*
C163	0.53600 (17)	0.18833 (14)	0.86345 (11)	0.0305 (3)
C164	0.68099 (18)	0.29526 (15)	0.91037 (12)	0.0343 (3)
F164	0.77811 (12)	0.29987 (10)	1.00385 (8)	0.0463 (2)
C165	0.72848 (19)	0.39468 (15)	0.86428 (13)	0.0380 (3)
H165	0.8275	0.4667	0.8982	0.046*
C166	0.63049 (19)	0.38888 (14)	0.76772 (13)	0.0350 (3)
H166	0.6627	0.4570	0.7350	0.042*
O163	0.50367 (13)	0.09665 (10)	0.91665 (9)	0.0379 (2)
C167	0.35126 (19)	−0.01095 (14)	0.87181 (13)	0.0370 (3)
H17A	0.3504	−0.0494	0.7936	0.055*
H17B	0.2559	0.0152	0.8765	0.055*
H17C	0.3431	−0.0722	0.9146	0.055*
S21	0.78227 (5)	0.13419 (4)	0.62262 (3)	0.03651 (11)
C22	0.94908 (17)	0.27885 (14)	0.68237 (12)	0.0330 (3)
N23	0.96304 (15)	0.36656 (12)	0.63253 (10)	0.0327 (3)
N24	0.83426 (14)	0.31918 (11)	0.54278 (9)	0.0300 (2)
C25	0.79000 (17)	0.36683 (13)	0.46012 (11)	0.0308 (3)
H25	0.8426	0.4488	0.4514	0.037*
C26	0.65212 (16)	0.26908 (13)	0.39250 (11)	0.0285 (3)
N27	0.61083 (14)	0.16220 (11)	0.43120 (10)	0.0311 (3)
C27A	0.72405 (17)	0.19776 (13)	0.52154 (11)	0.0299 (3)
C221	1.06817 (19)	0.29945 (17)	0.78443 (13)	0.0416 (4)
H22A	1.1227	0.2388	0.7704	0.062*
H22B	1.0074	0.2880	0.8459	0.062*
H22C	1.1530	0.3852	0.8043	0.062*
C261	0.55382 (16)	0.27107 (13)	0.29205 (11)	0.0282 (3)
C262	0.40568 (16)	0.16877 (13)	0.23951 (11)	0.0287 (3)
H262	0.3699	0.0984	0.2691	0.034*
C263	0.31052 (17)	0.16897 (13)	0.14477 (11)	0.0301 (3)
C264	0.36614 (18)	0.27413 (14)	0.10393 (12)	0.0337 (3)
F264	0.27138 (12)	0.27444 (9)	0.01147 (8)	0.0461 (2)
C265	0.51152 (19)	0.37496 (14)	0.15271 (12)	0.0349 (3)
H265	0.5468	0.4449	0.1225	0.042*
C266	0.60700 (17)	0.37331 (14)	0.24735 (12)	0.0327 (3)
H266	0.7091	0.4423	0.2817	0.039*
O263	0.16416 (13)	0.07545 (10)	0.08734 (9)	0.0384 (2)
C267	0.11887 (19)	−0.04054 (14)	0.11693 (13)	0.0373 (3)
H27A	0.0172	−0.1029	0.0660	0.056*
H27B	0.0982	−0.0270	0.1936	0.056*
H27C	0.2101	−0.0713	0.1115	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.03049 (18)	0.03356 (19)	0.03207 (18)	0.00582 (14)	0.00091 (13)	0.01204 (14)
C12	0.0296 (6)	0.0333 (7)	0.0292 (6)	0.0121 (6)	0.0071 (5)	0.0116 (5)
N13	0.0332 (6)	0.0335 (6)	0.0275 (6)	0.0119 (5)	0.0038 (5)	0.0138 (5)

N14	0.0299 (6)	0.0280 (6)	0.0279 (6)	0.0088 (5)	0.0048 (4)	0.0119 (4)
C15	0.0310 (7)	0.0308 (7)	0.0287 (6)	0.0086 (6)	0.0028 (5)	0.0108 (5)
C16	0.0286 (6)	0.0301 (6)	0.0269 (6)	0.0116 (5)	0.0058 (5)	0.0104 (5)
N17	0.0304 (6)	0.0322 (6)	0.0309 (6)	0.0093 (5)	0.0040 (5)	0.0123 (5)
C17A	0.0287 (6)	0.0288 (6)	0.0287 (6)	0.0100 (5)	0.0064 (5)	0.0104 (5)
C121	0.0330 (7)	0.0450 (8)	0.0337 (7)	0.0119 (6)	0.0018 (6)	0.0166 (6)
C161	0.0282 (6)	0.0333 (7)	0.0295 (6)	0.0145 (6)	0.0069 (5)	0.0118 (5)
C162	0.0280 (6)	0.0328 (7)	0.0279 (6)	0.0123 (5)	0.0039 (5)	0.0116 (5)
C163	0.0308 (7)	0.0364 (7)	0.0293 (7)	0.0164 (6)	0.0061 (5)	0.0124 (6)
C164	0.0317 (7)	0.0410 (8)	0.0300 (7)	0.0159 (6)	−0.0001 (5)	0.0079 (6)
F164	0.0418 (5)	0.0546 (6)	0.0377 (5)	0.0160 (4)	−0.0085 (4)	0.0136 (4)
C165	0.0307 (7)	0.0353 (7)	0.0406 (8)	0.0082 (6)	−0.0020 (6)	0.0067 (6)
C166	0.0334 (7)	0.0336 (7)	0.0376 (7)	0.0109 (6)	0.0034 (6)	0.0133 (6)
O163	0.0374 (6)	0.0410 (6)	0.0356 (5)	0.0121 (5)	−0.0012 (4)	0.0190 (5)
C167	0.0365 (8)	0.0382 (8)	0.0380 (8)	0.0121 (6)	0.0041 (6)	0.0183 (6)
S21	0.03315 (19)	0.0365 (2)	0.03448 (19)	0.00513 (15)	−0.00028 (14)	0.01498 (15)
C22	0.0257 (6)	0.0395 (7)	0.0295 (7)	0.0066 (6)	0.0033 (5)	0.0110 (6)
N23	0.0264 (5)	0.0362 (6)	0.0281 (6)	0.0055 (5)	−0.0020 (4)	0.0080 (5)
N24	0.0252 (5)	0.0301 (6)	0.0277 (6)	0.0042 (5)	0.0002 (4)	0.0065 (4)
C25	0.0279 (6)	0.0306 (7)	0.0296 (7)	0.0074 (5)	0.0005 (5)	0.0077 (5)
C26	0.0248 (6)	0.0313 (7)	0.0274 (6)	0.0096 (5)	0.0033 (5)	0.0067 (5)
N27	0.0269 (5)	0.0312 (6)	0.0299 (6)	0.0061 (5)	0.0012 (4)	0.0077 (5)
C27A	0.0260 (6)	0.0309 (7)	0.0285 (6)	0.0062 (5)	0.0036 (5)	0.0076 (5)
C221	0.0321 (7)	0.0519 (9)	0.0352 (8)	0.0074 (7)	−0.0020 (6)	0.0183 (7)
C261	0.0243 (6)	0.0321 (7)	0.0266 (6)	0.0118 (5)	0.0026 (5)	0.0044 (5)
C262	0.0254 (6)	0.0307 (7)	0.0288 (6)	0.0103 (5)	0.0021 (5)	0.0073 (5)
C263	0.0269 (6)	0.0318 (7)	0.0295 (6)	0.0122 (5)	0.0003 (5)	0.0046 (5)
C264	0.0321 (7)	0.0388 (8)	0.0316 (7)	0.0158 (6)	0.0011 (5)	0.0106 (6)
F264	0.0432 (5)	0.0505 (6)	0.0418 (5)	0.0130 (4)	−0.0072 (4)	0.0208 (4)
C265	0.0358 (7)	0.0352 (7)	0.0362 (7)	0.0138 (6)	0.0054 (6)	0.0147 (6)
C266	0.0264 (6)	0.0318 (7)	0.0361 (7)	0.0086 (6)	0.0019 (5)	0.0073 (6)
O263	0.0323 (5)	0.0346 (5)	0.0404 (6)	0.0066 (4)	−0.0094 (4)	0.0112 (4)
C267	0.0345 (7)	0.0304 (7)	0.0393 (8)	0.0065 (6)	−0.0051 (6)	0.0088 (6)

Geometric parameters (Å, º)

S11—C17A	1.7297 (14)	S21—C27A	1.7307 (15)
S11—C12	1.7605 (14)	S21—C22	1.7531 (15)
C12—N13	1.2970 (18)	C22—N23	1.3004 (19)
C12—C121	1.4849 (19)	C22—C221	1.4854 (19)
N13—N14	1.3717 (15)	N23—N24	1.3708 (15)
N14—C17A	1.3678 (18)	N24—C27A	1.3651 (18)
N14—C15	1.3687 (17)	N24—C25	1.3693 (18)
C15—C16	1.3740 (19)	C25—C26	1.3759 (18)
C15—H15	0.9500	C25—H25	0.9500
C16—N17	1.3978 (17)	C26—N27	1.3940 (18)
C16—C161	1.4630 (18)	C26—C261	1.4641 (18)
N17—C17A	1.3075 (18)	N27—C27A	1.3111 (17)

C121—H12A	0.9800	C221—H22A	0.9800
C121—H12B	0.9800	C221—H22B	0.9800
C121—H12C	0.9800	C221—H22C	0.9800
C161—C166	1.393 (2)	C261—C266	1.394 (2)
C161—C162	1.3991 (19)	C261—C262	1.3963 (18)
C162—C163	1.3862 (18)	C262—C263	1.3838 (18)
C162—H162	0.9500	C262—H262	0.9500
C163—O163	1.3559 (17)	C263—O263	1.3594 (16)
C163—C164	1.395 (2)	C263—C264	1.395 (2)
C164—F164	1.3581 (16)	C264—F264	1.3595 (16)
C164—C165	1.374 (2)	C264—C265	1.366 (2)
C165—C166	1.388 (2)	C265—C266	1.3912 (19)
C165—H165	0.9500	C265—H265	0.9500
C166—H166	0.9500	C266—H266	0.9500
O163—C167	1.4318 (18)	O263—C267	1.4291 (18)
C167—H17A	0.9800	C267—H27A	0.9800
C167—H17B	0.9800	C267—H27B	0.9800
C167—H17C	0.9800	C267—H27C	0.9800
C17A—S11—C12	88.62 (6)	C27A—S21—C22	88.44 (7)
N13—C12—C121	122.15 (13)	N23—C22—C221	121.98 (13)
N13—C12—S11	116.10 (10)	N23—C22—S21	116.53 (11)
C121—C12—S11	121.75 (11)	C221—C22—S21	121.49 (12)
C12—N13—N14	108.29 (11)	C22—N23—N24	107.87 (12)
C17A—N14—C15	107.58 (11)	C27A—N24—C25	107.60 (11)
C17A—N14—N13	118.81 (11)	C27A—N24—N23	118.89 (12)
C15—N14—N13	133.59 (12)	C25—N24—N23	133.45 (12)
N14—C15—C16	104.50 (12)	N24—C25—C26	104.29 (12)
N14—C15—H15	127.8	N24—C25—H25	127.9
C16—C15—H15	127.8	C26—C25—H25	127.9
C15—C16—N17	111.51 (12)	C25—C26—N27	111.80 (12)
C15—C16—C161	126.78 (13)	C25—C26—C261	126.64 (13)
N17—C16—C161	121.70 (12)	N27—C26—C261	121.56 (12)
C17A—N17—C16	103.55 (11)	C27A—N27—C26	103.30 (11)
N17—C17A—N14	112.86 (12)	N27—C27A—N24	113.01 (13)
N17—C17A—S11	138.95 (11)	N27—C27A—S21	138.68 (11)
N14—C17A—S11	108.17 (10)	N24—C27A—S21	108.27 (10)
C12—C121—H12A	109.5	C22—C221—H22A	109.5
C12—C121—H12B	109.5	C22—C221—H22B	109.5
H12A—C121—H12B	109.5	H22A—C221—H22B	109.5
C12—C121—H12C	109.5	C22—C221—H22C	109.5
H12A—C121—H12C	109.5	H22A—C221—H22C	109.5
H12B—C121—H12C	109.5	H22B—C221—H22C	109.5
C166—C161—C162	119.56 (13)	C266—C261—C262	119.44 (12)
C166—C161—C16	120.82 (13)	C266—C261—C26	120.87 (12)
C162—C161—C16	119.62 (12)	C262—C261—C26	119.68 (13)
C163—C162—C161	120.69 (13)	C263—C262—C261	120.61 (13)
C163—C162—H162	119.7	C263—C262—H262	119.7

C161—C162—H162	119.7	C261—C262—H262	119.7
O163—C163—C162	125.54 (13)	O263—C263—C262	125.73 (13)
O163—C163—C164	116.19 (12)	O263—C263—C264	116.04 (12)
C162—C163—C164	118.26 (13)	C262—C263—C264	118.22 (13)
F164—C164—C165	119.47 (13)	F264—C264—C265	119.48 (13)
F164—C164—C163	118.54 (13)	F264—C264—C263	118.02 (13)
C165—C164—C163	121.99 (13)	C265—C264—C263	122.50 (13)
C164—C165—C166	119.37 (14)	C264—C265—C266	118.80 (14)
C164—C165—H165	120.3	C264—C265—H265	120.6
C166—C165—H165	120.3	C266—C265—H265	120.6
C165—C166—C161	120.12 (14)	C265—C266—C261	120.40 (13)
C165—C166—H166	119.9	C265—C266—H266	119.8
C161—C166—H166	119.9	C261—C266—H266	119.8
C163—O163—C167	116.49 (11)	C263—O263—C267	116.68 (11)
O163—C167—H17A	109.5	O263—C267—H27A	109.5
O163—C167—H17B	109.5	O263—C267—H27B	109.5
H17A—C167—H17B	109.5	H27A—C267—H27B	109.5
O163—C167—H17C	109.5	O263—C267—H27C	109.5
H17A—C167—H17C	109.5	H27A—C267—H27C	109.5
H17B—C167—H17C	109.5	H27B—C267—H27C	109.5

C17A—S11—C12—N13	−0.34 (12)	C27A—S21—C22—N23	−0.59 (13)
C17A—S11—C12—C121	179.48 (12)	C27A—S21—C22—C221	179.60 (13)
C121—C12—N13—N14	−179.47 (12)	C221—C22—N23—N24	−179.25 (13)
S11—C12—N13—N14	0.35 (15)	S21—C22—N23—N24	0.94 (16)
C12—N13—N14—C17A	−0.18 (17)	C22—N23—N24—C27A	−0.95 (18)
C12—N13—N14—C15	−178.44 (14)	C22—N23—N24—C25	−177.64 (15)
C17A—N14—C15—C16	0.19 (15)	C27A—N24—C25—C26	0.17 (15)
N13—N14—C15—C16	178.59 (14)	N23—N24—C25—C26	177.13 (14)
N14—C15—C16—N17	−0.42 (16)	N24—C25—C26—N27	−0.27 (16)
N14—C15—C16—C161	−179.80 (13)	N24—C25—C26—C261	178.99 (13)
C15—C16—N17—C17A	0.47 (16)	C25—C26—N27—C27A	0.27 (16)
C161—C16—N17—C17A	179.89 (12)	C261—C26—N27—C27A	−179.04 (12)
C16—N17—C17A—N14	−0.34 (15)	C26—N27—C27A—N24	−0.15 (16)
C16—N17—C17A—S11	−178.18 (13)	C26—N27—C27A—S21	−177.30 (14)
C15—N14—C17A—N17	0.10 (16)	C25—N24—C27A—N27	−0.01 (17)
N13—N14—C17A—N17	−178.58 (11)	N23—N24—C27A—N27	−177.49 (12)
C15—N14—C17A—S11	178.61 (9)	C25—N24—C27A—S21	178.01 (9)
N13—N14—C17A—S11	−0.07 (15)	N23—N24—C27A—S21	0.53 (16)
C12—S11—C17A—N17	178.12 (17)	C22—S21—C27A—N27	177.26 (17)
C12—S11—C17A—N14	0.21 (10)	C22—S21—C27A—N24	0.02 (11)
C15—C16—C161—C166	−1.3 (2)	C25—C26—C261—C266	8.0 (2)
N17—C16—C161—C166	179.42 (13)	N27—C26—C261—C266	−172.79 (12)
C15—C16—C161—C162	178.65 (13)	C25—C26—C261—C262	−172.64 (13)
N17—C16—C161—C162	−0.7 (2)	N27—C26—C261—C262	6.6 (2)
C166—C161—C162—C163	0.3 (2)	C266—C261—C262—C263	−0.9 (2)
C16—C161—C162—C163	−179.58 (12)	C26—C261—C262—C263	179.71 (12)
C161—C162—C163—O163	−179.52 (13)	C261—C262—C263—O263	−179.49 (13)

C161—C162—C163—C164	−0.4 (2)	C261—C262—C263—C264	−0.4 (2)
O163—C163—C164—F164	0.3 (2)	O263—C263—C264—F264	−0.2 (2)
C162—C163—C164—F164	−178.97 (12)	C262—C263—C264—F264	−179.35 (12)
O163—C163—C164—C165	179.67 (14)	O263—C263—C264—C265	−179.62 (13)
C162—C163—C164—C165	0.4 (2)	C262—C263—C264—C265	1.2 (2)
F164—C164—C165—C166	178.95 (13)	F264—C264—C265—C266	179.91 (13)
C163—C164—C165—C166	−0.4 (2)	C263—C264—C265—C266	−0.6 (2)
C164—C165—C166—C161	0.4 (2)	C264—C265—C266—C261	−0.7 (2)
C162—C161—C166—C165	−0.3 (2)	C262—C261—C266—C265	1.5 (2)
C16—C161—C166—C165	179.57 (13)	C26—C261—C266—C265	−179.16 (13)
C162—C163—O163—C167	−3.5 (2)	C262—C263—O263—C267	−10.5 (2)
C164—C163—O163—C167	177.32 (13)	C264—C263—O263—C267	170.39 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···N13^{i}	0.95	2.61	3.551 (2)	169

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.